



Article

# Technological Analysis of the Production of Nickel-Containing Composite Materials

Bauyrzhan Kelamanov <sup>1</sup>, Dauren Yessengaliyev <sup>1,\*</sup>, Otegen Sariev <sup>1,\*</sup>, Askhat Akuov <sup>2</sup>, Yerulan Samuratov <sup>2</sup>, Talgat Zhuniskaliyev <sup>3</sup>, Yerbol Kuvatbay <sup>3</sup>, Yerbol Mukhambetgaliyev <sup>4</sup>, Olga Kolesnikova <sup>5,\*</sup>, Assel Zhumatova <sup>6</sup>, Zukhra Karaidarova <sup>6</sup> and Assylbek Abdirashit <sup>3</sup>

<sup>1</sup> Department of Metallurgy and Mining, K. Zhubanov Aktobe Regional University, Aktobe 030000, Kazakhstan; kelamanov-b@mail.ru

<sup>2</sup> Technical Development Department, Kazphosphate LLP, Taraz 080000, Kazakhstan; akuov.am@mail.ru (A.A.); samuratov.ek@mail.ru (Y.S.)

<sup>3</sup> Department of Metallurgy, Karaganda Industrial University, Temirtau 101400, Kazakhstan; talgat.zhuniskaliyev@mail.ru (T.Z.); kazakh\_84@mail.ru (Y.K.); a.abdirashit@tttu.edu.kz (A.A.)

<sup>4</sup> Laboratory of Steel Metallurgy and Materials Science, Abishev Chemical-Metallurgical Institute, Karaganda 100009, Kazakhstan; ye.kenzhegaliuly@gmail.com

<sup>5</sup> Department of Science, Production and Innovation, M. Auezov South Kazakhstan University, Shymkent 160012, Kazakhstan

<sup>6</sup> Department of Power Engineering, Satbayev University, Almaty 050013, Kazakhstan; a.zhumatova@satbayev.university (A.Z.); zuhrakaraidarova@gmail.com (Z.K.)

\* Correspondence: dauralga@mail.ru (D.Y.); rafhatsson@mail.ru (O.S.); ogkolesnikova@yandex.kz (O.K.); Tel.: +7-(7132)-54-06-19 (D.Y. & O.S.)

**Abstract:** The article presents the results of obtaining a composite material by sintering nickel-containing raw materials mixed with carbon-containing materials, namely using coke and semi-coke. The sintering process was performed at a charge layer height of 240 mm and the temperature of the lower layer was  $T = 1200$  °C. The results of the sieve analysis showed (a fraction of 10 mm) that the yield of a suitable composite material using coke was 68.3% and with semi-coke 67.0%. The average nickel and chromium content in the composite materials was 1.42% and 3.07%, accordingly. As a result of determining the strength characteristics of the obtained composite materials with various reducing agents by dropping from a height of 2 m onto a steel pallet, it was found that the obtained composite materials have high mechanical properties in terms of strength of 81% and 89.2%. The results of the elemental composition at the studied points and the thermal analysis of the studied composite material are presented. The mineralogical composition of the composite material is presented in the form of serpentine and nontronite, and the empty rock is made of quartz and talc. The activation energy of thermal analysis by the method of non-isothermal kinetics were calculated. The results of experiments on the production of composite materials from nickel-containing raw materials will be recommended for obtaining the optimal composition of composite materials at the stage of pilot tests and industrial development of the developed technology for processing nickel ores of the Republic of Kazakhstan. For the processing of nickel-poor nickel ores, it is of great importance to obtain optimal technological and technical and economic indicators that ensure low cost of nickel in the resulting product.

**Keywords:** nickel concentrate; nickel-containing composite materials; sintering process; thermographic studies; reduction kinetics; physico-chemical transformations; nickel-containing composite materials; alloys; environmental engineering; differential thermal analysis; activation energy



**Citation:** Kelamanov, B.; Yessengaliyev, D.; Sariev, O.; Akuov, A.; Samuratov, Y.; Zhuniskaliyev, T.; Kuvatbay, Y.; Mukhambetgaliyev, Y.; Kolesnikova, O.; Zhumatova, A.; et al. Technological Analysis of the Production of Nickel-Containing Composite Materials. *J. Compos. Sci.* **2024**, *8*, 179. <https://doi.org/10.3390/jcs8050179>

Academic Editor: Prashanth Konda Gokuldoss

Received: 20 April 2024

Revised: 5 May 2024

Accepted: 7 May 2024

Published: 12 May 2024



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## 1. Introduction

With the growth of production in ferrous metallurgy, the rational and efficient use of natural resources and reserves of industry, including improving the quality of metallurgical products and improving technology, is of great importance. Nickel-based alloys are no

exception, which are used in the production of high-quality grades of stainless, corrosion-resistant, heat-resistant, tool, structural and special-purpose steels, the industry of which is associated with the technological evolution of space technology, mechanical engineering, aviation, agriculture and energy. The main properties of nickel alloys are their use as alloying additives in the production of steel

When nickel is added to alloys of different metals, the properties of the alloys tend to improve. For example, the mechanical properties of ordinary steel, which is an alloy of iron with a small amount of carbon, manganese and flint, are significantly improved when nickel is added. Nickel, in addition to alloying steel, is used in chemistry, non-ferrous metallurgy and to a lesser extent for electrocoating. The addition of nickel to steel can improve its corrosion resistance, mechanical properties, impact strength, weldability and temperature resistance [1,2].

Improving the quality of steel, and as a result reducing metal consumption and improving the consumer properties of metal products, is one of the main and urgent problems of ferrous metallurgy.

One of the most important issues for ferrous metallurgy in the Republic of Kazakhstan is to provide it with nickel-containing alloys. In modern conditions, there is a clear tendency to significantly increase the proportion of corrosion-resistant steels. The annual consumption of nickel in the world has doubled over the past 20 years, which is associated with an increase in the production of nickel-containing steels and alloys. The basic alloying elements of stainless steels are chromium and nickel.

Metallurgical companies of the Republic of Kazakhstan do not experience a shortage of chrome ferroalloys; however, there is no effective technology for smelting nickel ferroalloys, and metallurgical enterprises in need of this ferroalloy are forced to purchase it abroad [3].

At the moment, due to the lack of their own production, metallurgical plants in the Republic of Kazakhstan are forced to import stainless steel, acid-resistant, heat-resistant, bearing and other steel marks. These steel marks are used as fittings for petroleum refineries, in high-pressure pumping units and for chemical industry devices where it contributes to the construction of new metallurgical plants of stainless steel marks [4,5].

In the modern metallurgical practice of the Republic of Kazakhstan, the processing of nickel ores is limited, which does not significantly increase their final cost. Nickel ores found in the depths of Kazakhstan are characterized by relative specificity and are represented by a number of powerful placer deposits: "Gornostaevskoye" (eastern region of the Republic of Kazakhstan), "Batamshinskoye", "Nikeltauskoye" (western region of the Republic of Kazakhstan). The main reserves of oxidized nickel ores are found in the Kempirsai group of silicate deposits located in the Aktobe region of the western part of Kazakhstan. At the moment, the total reserve of nickel ores in Kazakhstan is ~423.5 million tons [6]. However, the main reserves of nickel ores are substandard. Consequently, a serious direction is already being planned to increase the production of nickel-containing alloys from domestic nickel-containing raw materials. A further increase in the production of nickel-containing alloys according to accepted technologies will be associated with the involvement of increasingly poor nickel ores requiring deep enrichment. This will inevitably lead to an increase in losses and an increase in the cost of nickel-containing alloys. The way out of this situation, along with the intensification of prospecting and exploration of new deposits, is the parallel conduct of research on the development of effective technologies for processing nickel ores.

The problem of the involvement of oxidized nickel ores in metallurgical processing in Kazakhstan has not yet found a resolution. At this moment, despite many studies on the production of nickel-containing alloys, there are no technologies operating in industrial conditions in the country. On an industrial scale, technologies for the production of nickel casting, nickel matte and blast furnace cast iron at metallurgical plants of the Republic of Kazakhstan using nickel ores have not yet become widespread. Nickel-containing alloys are produced at foreign plants according to multi-stage processing schemes, including enrichment and agglomeration. Based on this, it is necessary to develop the most effective

technology for processing poor nickel ores from the Republic of Kazakhstan with subsequent production of nickel-based composite materials and smelting nickel-containing alloys from them.

As world practice shows, ores containing at least 1.2% Ni are processed for the production of ferronickel, and in nickel ores in Kazakhstan the Ni content is 0.4–1.11%, and these nickel ores mostly consist of small particles that are not suitable for electric melting.

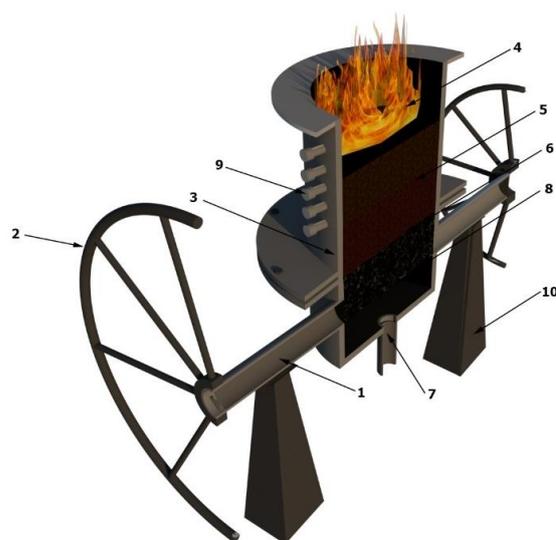
The goal of the work is to develop an effective technology for processing substandard nickel ores, which should include pre-oxidation of nickel ores with carbon-containing reducing agents to obtain a composite material.

## 2. Materials and Methods

The proven reserves of nickel deposits are able to provide metallurgical enterprises with raw materials for many years. In connection with this fact, there is an increasing interest in creating a technology for the production of nickel-based alloys using composite materials. In addition, when smelting nickel ores in ferroalloy furnaces, a significant amount of small and fine ore grains is removed by gases coming out of the furnace in the form of so-called grate dust. This leads to an increased specific consumption of charge materials and forces the processing of grate dust. Therefore, it is advisable to feed the charge materials into the furnace without a significant amount of fines (<5 mm), followed by the precipitation of fines. One of the rational ways to dip the small stuff of charge materials is the sintering process. The sintering process is a traditional and indispensable method of preparing metallurgical raw materials and helps to reduce energy consumption during smelting; the gas permeability of the charge materials is improved [7–13].

One of the most important methods of using low-quality metallurgical charge materials for the smelting of ferroalloys is the step-by-step preparation of raw materials for the production of composite materials. When crushing nickel-containing ore, a fine ore is formed [14,15].

To accomplish the set goal for sintering nickel ore with a diameter of 0–3 mm, research was conducted to obtain nickel-based composite materials in a laboratory installation. Figure 1 shows a schematic diagram technological scheme of an agglomeration plant for producing a nickel-based composite material.



**Figure 1.** Laboratory agglomeration equipment with a rotary mechanism. Key: 1—gas pipeline; 2—steering wheel; 3—bowl; 4—ignition mixture; 5—charge materials; 6—charge cake; 7—thermocouple; 8—grating for composite material production; 9—fitting; 10—support.

The research laboratory equipment with a rotary mechanism is equipped with an agglomeration table with a height of 350 mm and a diameter of 250 mm with a capacity of

up to 25 kg per experiment. Two types of reducing agent were used to study the sintering process. The chemical and technical compositions of the charge materials are shown in Table 1.

**Table 1.** Chemical and technical compositions of charge materials.

Material	Chemical Composition, %					
	Ni <sub>total</sub>	Fe <sub>total</sub>	Cr <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO
Nickel ore	1.11	15.88	2.25	47.99	1.23	5.19
Coke ash	-	10.25	-	40.59	14.61	6.08
Semi-coke ash	-	9.36	-	43.25	25.43	3.93
Technical composition, %						
Materials	C	V <sup>daf</sup> (volatility)	A <sup>c</sup> (ash)	W <sub>p</sub> (moister)		
Coke	85.24	2.70	10.11	1.32		
Semi-coke	87.24	2.11	7.96	2.13		

To conduct an experiment on the production of composite materials, we calculated the charge as follows:

First variant (kg): nickel ore—10; semi-coke—1.3 kg, return—2.1.

Second variant (g): nickel ore—20 kg; coke—1.5 kg; return—2.3. Mixtures of agglomerate and partially non-sintered charge fractions of 0–10 mm were used as a return. The moisture content of the charge was 6%. Before loading the charges into the agglomeration plant, they were crushed to 0–3 mm and mixed in a granulator.

Sintering procedure: The bed, heated to 60–80 °C, was loaded from the bowl, a ready-made charge was placed on top of it, then an ignition mixture was ignited after starting the vacuum pump, and a discharge was created under the grate of 200–300 mm of liquid column. By the end of the first minute, the flow rate was increased to 1000 mm of liquid column to ensure air suction through the charge layer using an exhaustor. The thermocouples were installed in the charge layer when loaded into the bowl. The next sintering was carried out after cooling the walls of the bowl to a temperature of 60–80 °C. The charge was ignited using a fuse and incendiary mixture for 1 min when discharged under a grate of 900–1000 mm of liquid columns. The sintering process proceeded intensively for an average of 25–30 min at a rate of 25.7–28.1 mm/min. Laboratory experiments were carried out at an average layer height of 240 mm. The sintering process was then carried out intensively with a flow rate of 1100–1200 mm of liquid column, while the layer temperature reached more than 1200 °C. The height of the charge layer was 340–350 mm.

To further study a number of properties that arise when sintering nickel ore with reducing agents to produce a nickel-containing composite material, we conducted research on these processes using the method of non-isothermal kinetics. As is known, when obtaining a new composite material, it is necessary to establish which physico-chemical transformations occur when it is heated at different temperatures. The main advantage of the non-isothermal method (in comparison with the isothermal method) is that many less experimental data are needed to calculate the kinetic parameters and the kinetic parameters can be calculated directly for the entire temperature range. One of the disadvantages of the non-isothermal method is the complexity of the hardware design, as well as the calculation of the kinetic parameters of the process. Currently, the most commonly used, widespread method of thermal analysis is differential thermal analysis (DTA). DTA with gradual heating of the substance under study makes it possible to identify and study phase transformations and chemical reactions in it, according to the thermal effects accompanying these changes. The results obtained make it possible to determine the rates of interaction reactions during the smelting of nickel alloys in industrial furnaces and select the most optimal composition of charge materials.

The *DTA* method is based on the most important properties of a substance related to its chemical composition and structure, which are reflected in the thermal changes in the substance during heating. Using *DTA*, thermogravimetry (*TG*) and differential thermogravimetry (*DTG*), new methods for studying solid-phase reactions when interacting substances are heated at a constant rate are being developed. The reason for the widespread use of non-isothermal kinetics methods is that all kinetic constants can be determined by one experiment:  $E_{act}$ ,  $A$  and  $n$ , where  $E_{act}$ —activation energy,  $A$ —pre-exponential multiplier (this coefficient is considered as intermolecular collisions),  $n$ —the general order of reaction. It should be noted that the reliability of the kinetic analysis carried out in this work, first of all, requires increased measurement accuracy and correct statistical processing of the results obtained. The literature describes various methods by which, after processing these curves (*TG* or *DTA*), the activation energy of processes can be determined [16–26].

Research on the studied materials was carried out on a derivatograph of the F. Paulik, J. Paulik, L. Erdei Derivatograph Q = 1000 system in the temperature range of 20–1400 °C with a heating step of 10 °C/min. The temperature in the furnace was measured by a platinum/platinum-rhodium thermocouple. As the temperature in the furnace of the derivatograph increases, the writing device registers on the thermogram all the physico-chemical changes occurring in the sample in the form of curved lines *T*, *TGA* and *DTA*. The temperature (*T*), the change in weight (*TGA*) and the rate of change in the thermal content (*DTA*) of the test substance are simultaneously measured in the sample depending on time.

The determination of kinetic parameters is the solution of a system of two equations relating the reaction rate constant  $k$  with temperature  $T$  and the reaction rate  $V$  with the degree of transformation  $\alpha$ :

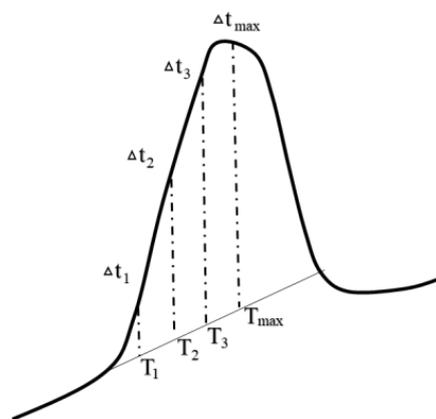
$$k = A \cdot \exp(-E_{act}/RT) \quad (1)$$

$$V = kf(\alpha) \quad (2)$$

where  $R$  is the gas constant.

For the case of decomposition of solids, the process rate is expressed by the equation  $V = k(1 - \alpha)^n$ , where  $n$  is the reaction order. The activation energy  $E_{act}$  is determined by the tangent of the slope angle of the dependence  $\lg = f(1/T)$ , obtained by the logarithm of the Arrhenius equation.

The value of the deviation of the differential record from the zero position with the apparent activation energy can be calculated by the tangent of the angle of inclination of the direct dependence of the deviation from the base line of the *DTA* curve in coordinates  $\lg = f(1/T)$ , that is, according to Figure 2, a dependence is built in coordinates  $\lg = f(1/T)$  for each thermal effect corresponding to peaks on the derivatogram [27–40].



**Figure 2.** Scheme for determining temperature values and the deviation of the *DTA* curve from a given direction.

### 3. Results

Figures 3 and 4 show images of composite materials obtained using various reducing agents. The sintered agglomerate using semi-coke had irregular or rounded pores formed as a result of uneven shrinkage of the charge during sintering and due to the release of gaseous compounds. When using coke, the agglomerate has a denser and less porous structure with a fine-grained structure of ore grains. The total weight of the nickel-containing composite material without minor changes was 970 g. Nickel composite materials had a denser and less porous structure with a fine-grained structure of ore grains. According to the measurement results according to GOST 25732-88, the true and apparent densities of the suitable composite material were 3.12 and 2.43 g/cm<sup>3</sup>, respectively, and the total porosity was 18.9–23.1%. The nickel content in the composite materials averaged 1.42%, while the nickel content in the initial ore was 1.11%. The ratio of nickel to iron averaged 0.07.



**Figure 3.** Nickel-containing composite material using nickel ore and semi-coke.



**Figure 4.** Nickel-containing composite material using nickel ore and coke.

To determine the suitability of the obtained material, nickel-containing composite materials were divided into size classes (+0–5 mm; +5–10 mm; +10 mm) obtained after sieve analysis. Figures 5 and 6 show the mass fractions and the distribution of elements by fractions of a nickel-containing composite material using various reducing agents. To study the strength properties of the obtained composite materials, the method of dropping from a height of 2 m (GOST 25471-82) onto a steel plate was applied. Table 2 shows the results of studies such as the mass fractions and the distribution of elements by fractions of a nickel-containing composite material after the experiment, that is, after a fall from a

height of 2 m. Table 3 shows the results of technological parameters of a composite material with various reducing agents.

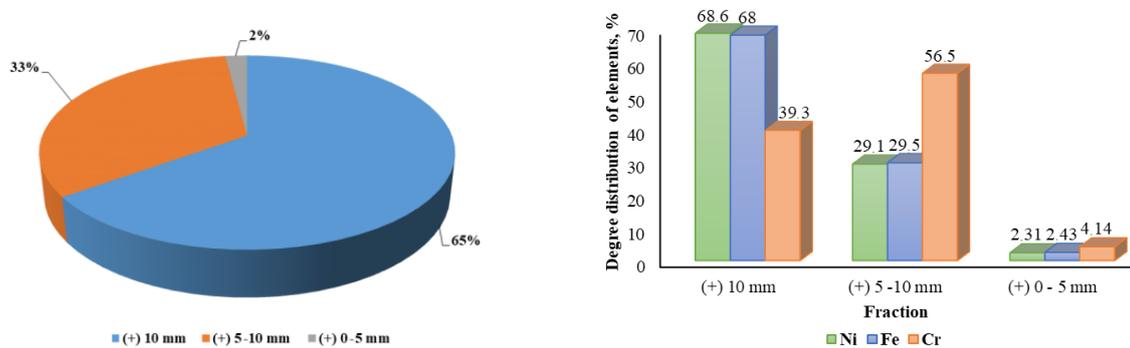


Figure 5. Mass parts and distribution of elements by fractions of nickel-containing composite material using nickel ore and semi-coke.

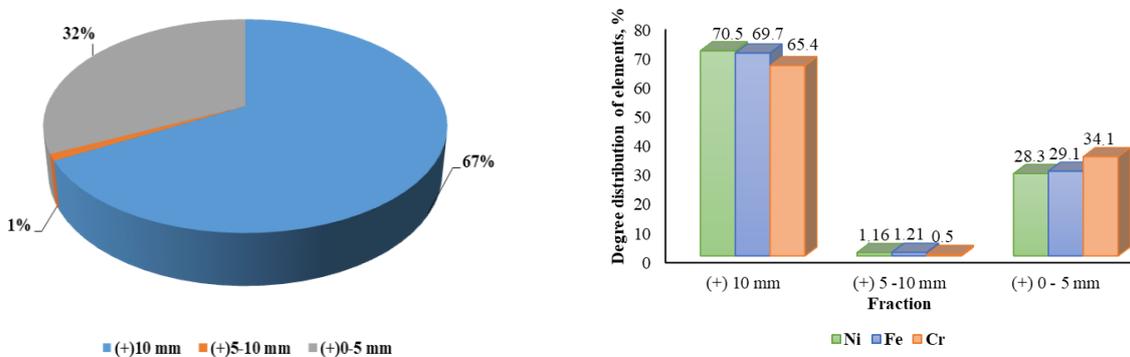


Figure 6. Mass parts and distribution of elements by fractions of nickel-containing composite material using nickel ore and coke.

Table 2. Chemical and fractional composition of composite materials.

Fraction, mm	The Yield of Suitable Products, %	Chemical Composition, %			Extraction, %		
		Ni <sub>total</sub>	Fe <sub>total</sub>	Cr <sub>total</sub>	Ni	Fe	Cr
Nickel-containing composite material using nickel ore and semi-coke							
(+) 10	75	1.44	18.20	1.94	75.0	74.5	48.0
(+) 5-10	6	1.45	19.00	6.88	6.0	6.20	13.0
(+) 0-5	19	1.44	18.50	6.24	19.0	19.0	40.0
Nickel-containing composite material using nickel ore and coke							
(+) 10	87	1.40	18.50	5.10	33.6	34.0	37.0
(+) 5-10	2	1.42	18.50	1.87	33.5	33.5	14.0
(+) 0-5	11	1.41	18.00	6.75	33.1	33.0	48.5

From the results presented in Tables 2 and 3, nickel composite materials, from the point of view of the yield of the useful fraction (+10 mm) and from the point of view of mechanical properties, can well serve as charge materials for smelting nickel-containing alloys.

**Table 3.** Technological parameters of a composite material with various reducing agents.

№	Parameters	Variants	
		Nickel-Containing Composite Material Using Nickel Ore and Semi-Coke	Nickel-Containing Composite Material Using Nickel Ore and Coke
1	Content in the charge materials,%		
	- semi-coke	10	-
	- coke	-	11
	- return	16	17
2	Wetness of charge materials, %	12–15	12–15
3	Layer height of charge materials, mm	350	350
4	Parameters of the sintering process		
4.1	- The maximum temperature in the layer of the charge material	1100	1100
4.2	- Shrinkage of charge materials, mm	55	55
4.3	- The output of a suitable product, (+ 10 mm)	67	68.3
5	The quality of the composite material		
	- Content, %		
5.1	Ni	1.46	1.38
	Cr	3.32	2.83
	Fe	19.0	18.3
5.2	- Strength of the composite material, % (+10 mm)	81.0	89.2

The laboratory-obtained composite material from nickel ore and coke has high strength characteristics compared to the composite material using semi-coke.

The maximum nickel composition in the sintering experiments was up to 1.45%, and the chromium composition was 6.88%. The resulting composite material in both cases had a high drop strength onto a steel plate. Thus, after dropping the tested composite materials from a height of 2 m onto a steel plate, it can be concluded that the output of a suitable composite material (+10 mm) using nickel ore with semi-coke was—67%. The output of a suitable composite material (+10 mm) using nickel ore with coke was 68.3%; in addition, the strength of the materials was 81% and 89%, respectively.

Table 3 shows that the nickel and chromium content in the composite material is sufficient to produce nickel-chromium-containing alloys. Based on the properties of the obtained nickel composite materials, in order to prevent its destruction, it is advisable to carry out production at the place of use, directly at ferroalloy plants.

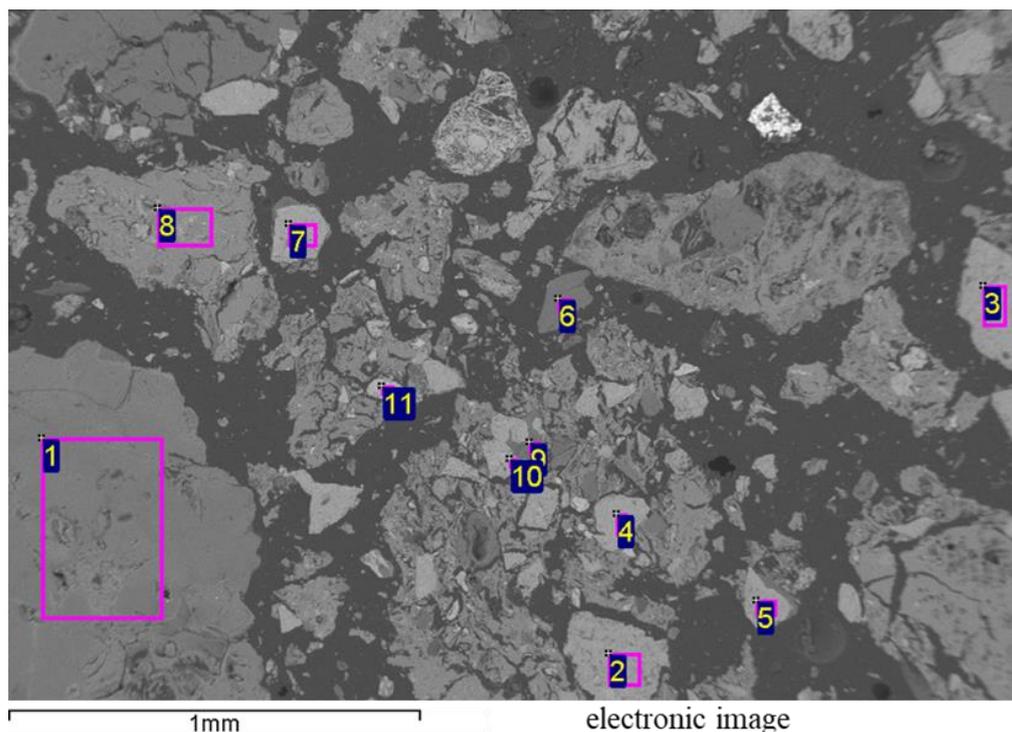
#### 4. Discussion

Nickel ores are characterized by great heterogeneity, and in some cases by the variability of their chemical composition. Many experiments on the enrichment of silicate ores have not yielded positive results to date. It can be seen from the results of the enrichment of silicate ores that silicate ores are not suitable for enrichment in terms of their physico-chemical properties, as well as in terms of the distribution of nickel in the ore. In silicate ores, nickel compounds are not found in the form of individualized and separate minerals, but in the form of a thin layer of hydrosilicate and other minerals in the entire mass of primary decomposition products of ultrabasic rocks. These minerals are often found at the site of contact of serpentinites with limestones and are accompanied by very fine clay rocks and hydrated iron oxides. Therefore, these ores are characterized as an earthy material, similar to clay.

Such a physico-chemical state of silicate nickel ores makes it impossible to open individual minerals containing nickel, and, consequently, implies the impossibility of their

separation and enrichment. Nickel ores of the Republic of Kazakhstan need preliminary preparation for metallurgy.

As is known, when obtaining a new nickel-containing material, it is necessary to study the physico-chemical state of the material. In order to fully study the properties of composite materials, we conducted studies to establish the structural data of the composite material and provided cartographic data on the distribution of elements. According to the research results, the structural composition of the composite material with various reducing agents has the same structure. Therefore, only one type of composite material is indicated in Figure 7.



**Figure 7.** Structural elements of the composite material sample under research.

To study samples of nickel-containing composite material using optical and electron microscopes, they were filled with epoxy resin, sanded and polished. The chemical composition of the structural elements of the samples was determined using a JEOL JSM-6460LV electron microscope using an energy dispersion analyzer from Oxford Instruments to determine the elemental composition at points and by area using the microrentgenospectral method. The chemical composition of the composite material obtained using the JSM-6460LV electron microscope is shown in Table 4.

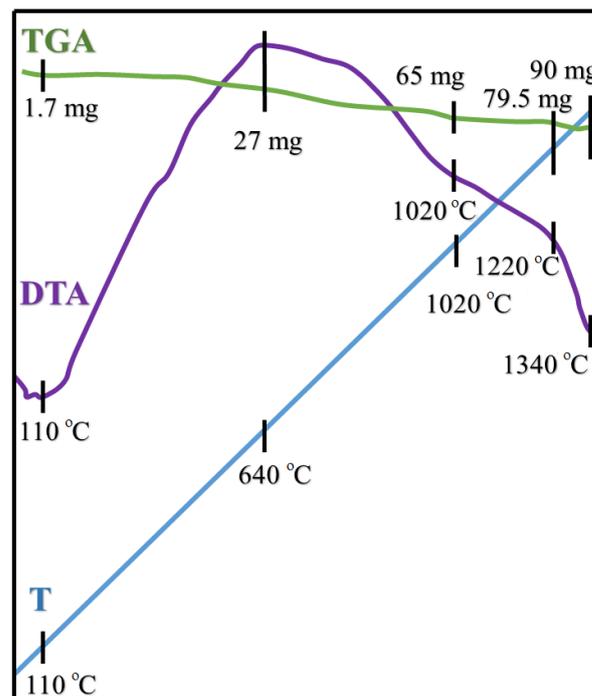
Studies have established that the mineralogical and elemental composition of nickel-containing composite materials is mainly represented by the minerals serpentine (antigorite)  $3\text{MgO}\cdot 2\text{SiO}_2\cdot 2\text{H}_2\text{O}$  and nontronite  $(\text{Fe,Al})_2[\text{Si}_4\text{O}_{10}(\text{OH})_2\cdot n\text{H}_2\text{O}]$ ; silicon-containing phases are present mainly in the form of opal ( $\text{SiO}_2$ ).

The mineral part of nickel ore is present in the form of hydrosilicates and aluminosilicates of nickel (garnierite, revdinskite, etc.). Magnesia-nickel hydrosilicates and hydro-aluminosilicates, as well as occasionally occurring hydrates and other nickel inclusions, are represented in small quantities in the composite material. There are practically no separate large-sized nickel-containing minerals in the ore part. Quartz and talc, in the form of clay, are present in the empty waste.

**Table 4.** Chemical composition of the composite material obtained using the JSM-6460LV electron microscope.

Spectrum	O	Ni	Mg	Al	Si	Ca	Cr	Mn	Fe
1	61.5	0.7	17.0	-	18.1	0.1	0.1	-	2.5
2	60.5	-	9.9	-	2.9	0.1	19.6	0.2	4.1
3	57.7	-	9.0	-	0.8	-	23.7	0.2	5.4
4	60.4	-	8.9	-	-	0.1	22.8	0.2	4.9
5	60.2	-	9.3	-	-	0.1	23.2	0.2	4.3
6	59.0	-	23.2	-	16.5	-	0.3	-	1.0
7	58.2	-	9.0	-	0.6	-	24.5	0.3	4.6
8	58.7	1.1	2.4	2.0	21.4	0.4	0.1	2.5	9.3
9	57.3	0.2	20.3	4.3	14.8	0.1	2.3	-	0.7
10	59.0	-	10.6	-	0.6	-	24.0	0.2	3.2
11	61.5	0.2	-	2.1	4.3	0.1	0.2	-	30.6

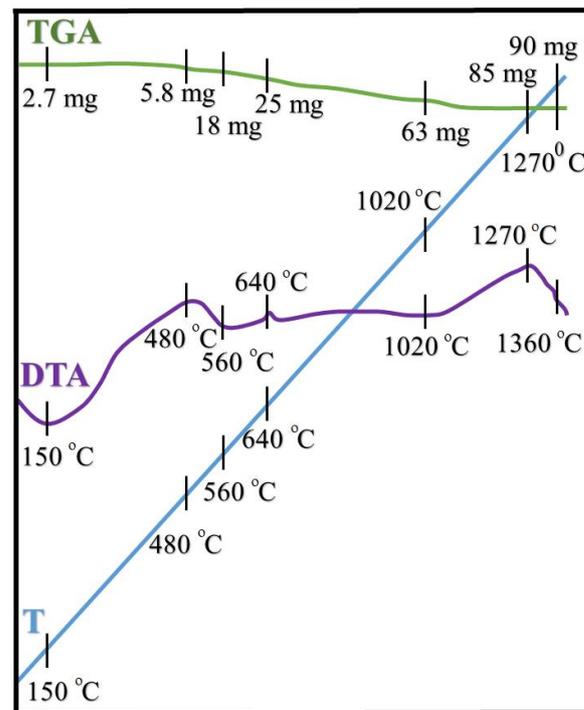
Figures 8 and 9 show the research data in the form of a derivatogram of nickel-containing composite materials using semi-coke and coke.



**Figure 8.** Results of a derivatogram of a nickel-containing composite material using nickel ore and semi-coke.

DTA found that nickel-containing composite materials have four transitions. When the temperature  $T = 110\text{ }^{\circ}\text{C}$  is reached, the first endothermic peak appears, accompanied by the deletion of hygroscopic moisture from the coal with a mass loss of 1.70 mg. The second exothermic effect is formed at a temperature of  $T = 640\text{ }^{\circ}\text{C}$ , where the release of hydrated moisture and the removal of volatile components is completed. The weight loss at this temperature was 27 mg. At a temperature range of  $T = 500\text{--}600\text{ }^{\circ}\text{C}$ , physico-chemical changes occur in the sample with the decomposition of serpentine ( $3\text{MgO}\cdot 2\text{SiO}_2\cdot 2\text{H}_2\text{O}$ ) and siderite ( $\text{FeCO}_3$ ). Further, with an increase in temperature, the process of interaction of the

nickel-containing composite material with the reducing agent occurs. At a temperature of 1020 °C, a third weakly expressed endothermic effect is observed with a decrease in sample mass by a total of 65 mg. This endothermic effect is explained by the complete combustion of solid carbon. When the material was heated to 1220 °C, a fourth exothermic effect was recorded, characterized by the appearance and establishment of the main components of natural minerals ( $(\text{Fe,Al})_2 [\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$ ;  $\text{Fe}_2\text{O}_3$ ;  $\text{FeCr}_2\text{O}_4$ ). The total weight loss at this temperature was 79.5 mg, and the total weight loss in the studied nickel-containing composite material was 90 mg.



**Figure 9.** The results of a derivatogram of a nickel-containing composite material using nickel ore and coke.

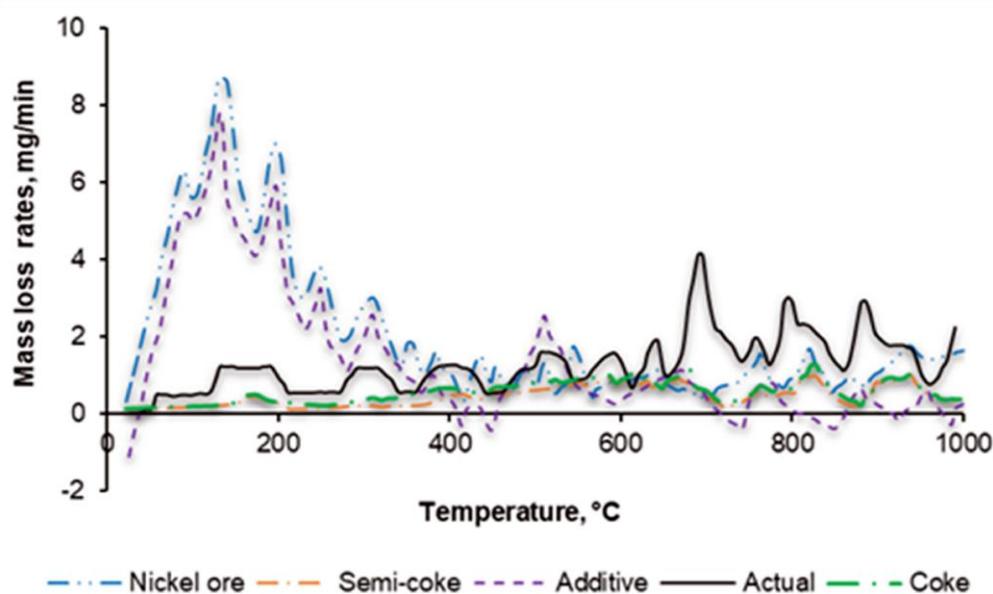
The curve line in the accident of the composite material is similar to the previous derivatogram. As shown in Figure 9, the first endothermic effect was observed at a temperature of 150 °C with a weight loss of 2.7 mg. This effect describes the complete removal of hygroscopic moisture from coke. Further, at temperatures of 480 °C and 560 °C, the second exothermic and third endothermic effects were manifested, corresponding to the combustion of volatile substances and the decomposition of the serpentine mineral ( $3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ). In addition to these transformations, decomposition of the siderite mineral  $\text{FeCO}_3$  can be observed at a temperature of 560 °C. The total weight loss at these temperatures was 5.8 mg and 18 mg, respectively. The fourth exothermic effect at a temperature of 640 °C leads to the beginning of recovery processes with a decrease in total weight by 25 mg. A further increase in temperature to 1020 °C shows the formation of a fifth weakly pronounced endothermic effect with a total weight loss of 63 mg. Then the appearance of a sharp maximum at a temperature of 1270 °C (the sixth exothermic effect) indicates the partial formation of the main phases of minerals ( $(\text{Fe, Al})_2 [\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$ ;  $\text{Fe}_2\text{O}_3$ ;  $\text{FeCr}_2\text{O}_4$ ) with a decrease in mass by 85 mg. The total mass loss of this composite material was 90 mg.

When considering the derivatogram curves of nickel-containing composite material, as noted above, there are a number of endothermic and exothermic effects, which made it possible to identify the most characteristic temperature ranges. The analysis of mass loss determination and temperature difference is shown in Table 5.

**Table 5.** Distribution of mass loss in composite materials in specified temperature ranges in accordance with the derivatogram.

Temperature, °C	Weight Loss, mg									
	Nickel Ore		Semi-Coke		Coke		Additive		Actual	
	mg	%	mg	%	mg	%	mg	%	mg	%
0–200	92.49	59.00	3.21	6.31	3.28	6.20	95.69	46.1	5.50	8.73
200–560	45.46	29.00	13.61	26.77	15.02	28.4	59.07	28.45	15.50	24.60
560–800	12.54	8.00	16.89	33.23	17.21	32.54	29.44	14.18	22.00	34.92
800–1000	6.27	4.00	17.13	33.69	17.37	32.84	23.4	11.27	20.00	31.75
0–1000	156.76	100.0	50.84	100.0	52.88	100.0	207.6	100.0	63.00	100.0
Hitch weight	940.56	100.0	59.44	100.0	60.12	100.0	100.0	100.0	1000	100.0

To compare the mass loss rates of the actual and additive mixtures, the curves (Figure 10) of the mass loss rates of ore, coke and their additive and actual mixtures (obtained by processing the TG curve) were compared, a comparison of which shows that the maximum mass loss rate for the actual and additive mixtures is shifted towards lower temperatures 20–200 °C.



**Figure 10.** Mass loss rates for a mixture of nickel ore and semi-coke during continuous heating at a given speed (10 °C/min) in an air atmosphere.

As can be seen from Table 4 and Figure 10, the mass loss of the additive mixture of ore and semi-coke exceeds the mass loss of the actual mixture of ore and coke in the temperature range 0–600 °C, which indicates a low development of physico-chemical processes of dehydration and the slow passage of the reduction process during continuous heating; only at temperatures above 600 °C the weight loss of the actual composite material mixture shows the reverse nature of the process. From Figure 10, it can be seen that the maximum weight loss of the composite material is in the region of low temperatures 20–200 °C, which is explained by the removal of natural moisture. The exothermic peaks are especially large in the temperature range of 700–800 °C, which corresponds to the onset of carbon combustion and decomposition of the nontronite mineral  $(Fe,Al)_2 [Si_4O_{10}(OH)]_2 \cdot nH_2O$ .

Using the technique shown in Figure 2, the values of  $\log \Delta t$  and  $1/T$  were determined for each thermal effect corresponding to the peaks on the derivatogram (Tables 6 and 7).

Based on the results of the  $\lg\Delta t$  and  $1/T$  values presented in Tables 6 and 7, the  $E_{act}$  activation energy values for each thermal effect are calculated. The results of the  $E_{act}$  indicator are shown in Table 8. The observed decrease in the index of  $E_{act}$  peak individually is associated with the influence of the reducing agent on the ongoing processes, i.e., the energy barrier decreases and the sintering process accelerates to obtain a homogeneous composite material. Thus, the results obtained make it possible to navigate the rate of interaction reactions during the smelting of nickel-containing alloys in industrial furnaces.

**Table 6.** The parameter values of  $\Delta t, T, \lg\Delta t, 1/T$  for calculating the apparent activation energy by the tangent of the angle of inclination of the direct dependence for a nickel composite material (ore+ semi-coke) (Figure 8).

No.	Temperature, °C	№	$\Delta t, \text{mm}$	$t, \text{°C}$	T, K	$\lg\Delta t$	$1/T$
1	110	0	0	10	283	-	-
		1	4	30	303	0.60206	0.0033
		2	27	50	323	1.431364	0.003096
		3	45	70	343	1.653213	0.002915
		4	52	90	363	1.716003	0.002755
		5	55	110	383	1.740363	0.002611
2	640	0	0	520	793	-	-
		1	2	540	813	0.30103	0.00123
		2	5	560	833	0.69897	0.0012
		3	8	580	853	0.90309	0.001172
		4	14	600	873	1.146128	0.001145
		5	15	620	893	1.176091	0.00112
3	1020	6	16	640	913	1.20412	0.001095
		0	0	920	1193	-	-
		1	4	940	1213	0.60206	0.000824
		2	6	960	1233	0.778151	0.000811
		3	8	980	1253	0.90309	0.000798
		4	12	1000	1273	1.079181	0.000786
4	1220	5	14	1020	1293	1.146128	0.000773
		0	0	1040	1313	-	-
		1	3	1060	1333	0.477121	0,00075
		2	9	1080	1353	0.954243	0.000739
		3	13	1100	1373	1.113943	0.000728
		4	16	1120	1393	1.20412	0.000718
		5	18	1140	1413	1.255273	0.000708
		6	22	1160	1433	1.342423	0.000698
		7	25	1180	1453	1.39794	0.000688
		8	28	1200	1473	1.447158	0.000679
9	30	1220	1493	1.477121	0.00067		

**Table 7.** The parameter values of  $\Delta t$ ,  $T$ ,  $\lg \Delta t$ ,  $1/T$  for calculating the apparent activation energy by the tangent of the angle of inclination of the direct dependence for a nickel composite material (ore+ coke) (Figure 9).

No.	Temperature, °C	№	$\Delta t$ , mm	$t$ , °C	T, K	$\lg \Delta t$	1/T
1	150	0	0	30	303	-	-
		1	17	50	323	1.230449	0.003096
		2	41	70	343	1.612784	0.002915
		3	55	90	363	1.740363	0.002755
		4	65	110	383	1.812913	0.002611
		5	73	130	403	1.863323	0.002481
		6	80	150	423	1.90309	0.002364
2	480	0	0	360	633	-	-
		1	2	380	653	0.30103	0.001531
		2	4	400	673	0.60206	0.001486
		3	7	420	693	0.845098	0.001443
		4	12	440	713	1.079181	0.001403
		5	15	460	733	1.176091	0.001364
		6	17	480	753	1.230449	0.001328
3	560	0	0	480	753	-	-
		1	1	500	773	0	0.001294
		2	6	520	793	0.778151	0.001261
		3	15	540	813	1.176091	0.00123
		4	17	560	833	1.230449	0.0012
4	640	0	0	600	873	-	-
		1	1	610	883	0	0.001133
		2	2	620	893	0.30103	0.00112
		3	4	630	903	0.60206	0.001107
		4	5	640	913	0.69897	0.001095
5	1020	0	0	960	1233	-	-
		1	2	970	1243	0.30103	0.000805
		2	4	980	1253	0.60206	0.000798
		3	7	990	1263	0.845098	0.000792
		4	12	1000	1273	1.079181	0.000786
		5	15	1010	1283	1.176091	0.000779
		6	17	1020	1293	1.230449	0.000773

**Table 8.** The results for activation energy  $E_{act}$ , determined by  $\log \Delta t - 1/T$ .

No.	Material	Equation	Correlation Coefficient R	$E_{act}$ , kj/mol	Temperature Interval, °C
1	Nickel-containing composite material using nickel ore and semi-coke	$\ln \Delta t = -154.33/T + 5.95$	0.8803	2.955	10–110
		$\ln \Delta t = -664.39/T + 8.61$	0.9446	12.721	520–640
		$\ln \Delta t = -1091.2/T + 9.61$	0.9932	20.895	920–1020
		$\ln \Delta t = -1039.1/T + 8.54$	0.9103	19.896	1040–1220

Table 8. Cont.

No.	Material	Equation	Correlation Coefficient R	E <sub>act</sub> , kJ/mol	Temperature Interval, °C
2	Nickel-containing composite material using nickel ore and coke	$\ln\Delta t = -84.43/T + 3.97$	0.9279	1.616	30–150
		$\ln\Delta t = -468.31/T + 7.54$	0.9781	8.967	360–480
		$\ln\Delta t = -1327.3/T + 17.34$	0.9377	25.422	480–560
		$\ln\Delta t = -1936.7/T + 21.97$	0.9807	37.083	600–640
		$\ln\Delta t = -3040.8/T + 24.85$	0.9716	58.225	960–1020
		$\ln\Delta t = -1162.3/T + 9.43$	0.8889	22.255	1050–1270

## 5. Conclusions

1. Laboratory experiments on agglomeration of nickel ores were carried out. For agglomeration of nickel ores, the most common method of charge preparation in metallurgy is used—agglomeration. In all experiments on the production of nickel-containing composite material, the results were satisfactory: the output of a suitable composite material in terms of size +10 mm was 67–68%, with a strength of 81–89%. Consequently, the resulting composite material with these characteristics can be suitable for use as a charge for ore-thermal furnaces;

2. As a result of research using the method of differential thermal analysis, it was established that the physico-chemical processes occurring when heating nickel-containing composite material with various reducing agents are more developed, which is demonstrated by the dependence of the actual rate of mass loss obtained by processing the TG curves of derivatogram;

3. The activation energy of processes occurring during heat treatment of nickel-containing composite material using the method of non-isothermal kinetics was determined. Determination of activation energy allows obtaining data on kinetic parameters, flowing during sintering of nickel ores, especially data on the recovery rates of the materials under study. That is, the parameters characterizing the exponential dependence of the reaction rate were constant on temperature;

4. The analysis of temperature peaks and activation energy levels of processes accompanied by peaks on the DTA curves of nickel-containing composite materials suggests that diffusion processes occurring during heat treatment and responsible for solid-phase hardening of sintered materials occur under more favorable conditions. Therefore, we can conclude that the processes occurring during the heat treatment of nickel-containing composite materials proceed at a fairly high speed and a high degree of completion is achieved at sintering temperatures of 600–1000 °C.

**Author Contributions:** B.K.: Guidelines and definition of research strategy; writing the article. D.Y.: Explanation of the theoretical foundations of the processes; organization of scientific experiments. O.S.: Planning of scientific experiments; organization of technological calculations. A.A. (Askhat Akuov) and Y.S.: Conducting laboratory experiments; analyzing the results obtained. T.Z. and Y.K.: work with reagents, materials, tools; participation in experimental studies. Y.M.: Analysis and interpretation of the obtained data; participation in experimental studies. All authors participated in writing the paper. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research is funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP 19679501).

**Data Availability Statement:** The data used to support the findings of this study are included within this article.

**Conflicts of Interest:** The authors declare no conflicts of interest.

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